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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Appln. of: OKADA et al.
Serial No.: 09/913,625
Filed: AUGUST 16, 2001
For: HYDROGEN STORAGE METAL ALLOY AND METHOD...
Group: 1742
Examiner: IP, SIKYIN DOCKET: SHIG C11119

Commissioner for Patents
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DECLARATION UNDER 37 CFR 1.132

The undersigned, being the named inventors of the above identified Application, declare and state as follows:

- (1) This Declaration is being filed in further support of our above identified U.S. Application. Our CV's are attached.
- (2) We understand our Application has been rejected as being obvious from Japanese Application No. JP 10-121180 (JP '180), taken alone or in combination with JP 11-106859. In particular, we understand the Examiner has cited JP '180 as teaching the advantages of quenching in iced water. We respectfully disagree. While JP '180 may mention quenching in iced water, nowhere within the four corners of JP '180 do the authors teach the advantages of quenching in iced water. Indeed, the working Examples in JP '180 do not use iced water.
- (3) Quenching in iced water is critical to the practice of our invention. To demonstrate the criticality of quenching in iced water, we conducted a series of experiments

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comparing the results of oil-quenching and quenching in iced water after heat treatment. The results are reported in Graphs 1 and 2 attached hereto, wherein:

Graph 1 shows X-ray diffraction patterns of Ti-57.5Cr-2.5 Mo alloys (oil-quenched or iced water-quenched after heat treatment). From Graph 1 it is shown that the iced water-quenched alloy has only clear BCC peaks while an oil-quenched alloy is contaminated with C15 & C36 Laves.

Graph 2 shows PTC curves of Ti-57.5Cr-2.5Mo alloys oil-quenched or iced water-quenched after heat treatment). From Graph 2, it is shown that the iced water-quenched alloy provides higher protium concentration (mass%) values, about 3.4 to 3.5 mass%, than an oil-quenched alloy.

(4) We have discovered that "rapidly cooled preferably at a cooling rate of more than 100K/sec." (page 19, lines 31 to 32 of the Applicants' sworn specification) is critical. The hydrogen storage alloys of the present invention have unexpectedly higher hydrogen storage capacities (see FIGS. 9 & 10). In FIGS. 9 & 10 of the subject Application, each storage capacity of the instant alloys is over 2.6 mass% within a range of both $0 < \text{Mo at\%} < 5$ and $0 < \text{W at\%} < 5$.

(5) A difference of 0.1 to 0.2 mass% per unit alloy mass is a remarkable value in this field in connection with Ti-Cr-system alloys having a quite low level of Mo and/or W. The increase of even 0.1 to 0.2 mass % in the hydrogen storage capacity is an unexpected, superior effect over the prior art alloys. This difference is derived from the difference between prior art

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alloy treatment processes and the instant unique ones, and reflects the greatly different lattice structures (i.e., highly BCC-rich structures).

(6) There are other distinctions. JP '180 fails to teach or suggest a "homogenizing" treatment as required by step (2) of claim 22 of the present invention. The prior art technical concept with faults leads to "The preferred range of Mo and/or W is 5% or more but 20% or less" ([0012] on page 3, column 4, lines 8 to 10 of JP '180).

(7) Also critical to our invention is the discovery that the highly elevated temperature is effective in forming BCC-rich alloys and the heat treatment, i.e. of 1400°C or higher, has to carry out within a very short period of time. In the working Examples of Applicants' sworn specification, all the alloys are produced at 1400°C or higher. As demonstrated in the attached Graphs 3 and 4, a short time heat treatment is preferable:

Graph 3 shows hydrogen absorption and desorption characteristics of Ti-57.5 Cr-2.5 Mo alloy where the heat-treatment time varies at 1400°C; and

Graph 4 shows PTC curve profiles of Ti-57.5 Cr-2.5 Mo alloy where the heat-treatment time varies at 1400°C.

From Graphs 3 and 4 it is demonstrated that the prolonged heat-treatment deteriorates the hydrogen absorption and desorption characteristics of hydrogen storage alloy products. See also Cr-Mo-Ti state diagrams, Graph 5.

(8) We have found that it is extremely important to:

(1) repeatedly melt and solidify the starting mixture to form a heated homogeneous alloy,

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(2) maintain the resultant heated homogeneous alloy at a temperature within a range just below the melting point of the alloy (i.e., 1400°C or higher) for a predetermined time (rather a shorter period) of from 1 to 100 minutes, and

(3) rapidly cooling the alloy from the above step (b) in iced water, and the combination of the above steps results in the formation of unexpected BCC-rich (or high hydrogen storage capacity) alloys even at a quite low level of Mo and W.

(9) Thus, it is shown that the combination of repeatedly melting and solidifying the starting mixture to form a heated homogeneous alloy, maintaining the heated homogeneous alloy at a temperature above 1400°C for from 1 to 100 minutes, and rapidly cooling the alloy in iced water results in hydrogen storage alloys having unique properties not achievable by the prior art.

(10) We hereby declare that all statements made hereof of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: Jan. 8, 2004

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